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Attorney Docket No:0492611-0477/ MIT-10051
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:	Jensen, et al.	Examiner:	LEUNG, JENNIFER A
Serial No.:	10/626,436	Art Unit:	1764
Filing Date:	07-24-2003		
Title:	**MICROCHEMICAL METHOD AND APPARATUS FOR SYNTHESIS AND COATING OF COLLOIDAL NANOPARTICLES**		

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION UNDER 37 C.F.R. § 1.131

Klavs F. Jensen declares as follows:

1. I am a professor of chemical engineering at the Massachusetts Institute of Technology in Cambridge Massachusetts.

2. I am a co-inventor of the invention disclosed and claimed in the above identified patent application. In particular, the invention relates to microchemical systems for the synthesis and coating of colloidal nanoparticles.

3. All of the events set forth in this declaration occurred in the United States of America.

4. I am submitting this declaration to establish completion of the invention disclosed and claimed in the above identified patent application in this country prior to February 26, 2003, the earliest priority date of published United States Patent Application No. U.S. 2005/0129580 A1 to Swinehart, et al.

5. Prior to February 26, 2003 my co-inventor and I conceived of the invention disclosed and claimed in the above identified patent application. Thereafter, and also prior to February 26, 2003 we reduced the invention to practice.

6. We filled out a Massachusetts Institute of Technology Technology Disclosure form and attached to it a description of the conception and actual reduction to practice of the invention.

7. Exhibit A is a copy of the Technology Disclosure form and attached description entitled "Microchemical Systems for Synthesis and Coating of Colloidal Nanoparticles" submitted to the MIT Technology Licensing Office on a date prior to February 26, 2003. The original of the Technology Disclosure form includes dates of conception, reduction to practice, signature dates and a date forming part of a "received" stamp. All of these

dates are prior to February 26, 2003 and have been redacted in Exhibit A. The original of the attached description also includes dates on each page as part of a "received" stamp. These dates are prior to February 26, 2003 and have also been removed.

8. Exhibit A provides evidence of conception and actual reduction to practice. Note that Fig. 2 in Exhibit A shows TiO_2 particles obtained by means of the microreactor shown in Fig. 1.

9. After submitting the Technology Disclosure form to the MIT Technology Licensing Office, the Technology Licensing Office, on a date prior to February 26, 2003 engaged attorney Bo Pasternack of the firm of Choate, Hall & Stewart to prepare and file a utility patent application.

10. Exhibit B is a letter from the MIT Technology Licensing Office to Mr. Pasternack initiating the patenting process. The original of the letter of Exhibit B included a date prior to February 26, 2003.

11. Thereafter, I reviewed the patent application and was informed that the patent application was filed on July 24, 2003.

12. I further declare that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true. I also understand that willful false statements and the like are punishable by fine of imprisonment or both under 18 U.S.C. 1001 and may jeopardize the validity of the application or any patent issuing thereon.

Respectfully submitted,

Klavs F. Jensen

Date: January 12, 2007

When completed submit via:
Technology Licensing Office
Room NE25-230
617-253-6966

Massachusetts Institute of Technology
TECHNOLOGY DISCLOSURE

Case No. (this space for TLO use only) 10051

Instructions on reverse

1. TITLE OF INVENTION
MICROCHEMICAL SYSTEMS FOR SYNTHESIS AND COATING OF COLLOIDAL NANOPARTICLES

2. PLEASE ATTACH DESCRIPTION OF TECHNOLOGY

3. INVENTOR(S)	POSITION	DEPARTMENT	M.I.T. ROOM NO. & EXTN.
KLAVS F. JENSEN	PROFESSOR	CHEMICAL ENGINEERING	66-566, x3-4589
SAIF A. KHAN	GRADUATE STUDENT	CHEMICAL ENGINEERING	66-469, x3-6654

4. Was this invention developed with the use of any research grant/contract funds? YES ☑ NO ☐

~~CONTRACT NO(S).~~	SPONSOR(S)	O.S.P. PROJECT NO(S).	PRINCIPAL INVESTIGATOR
6891693 (OSP)	CONSORTIUM		Klavs F. Jensen

Please note that accurate and complete sponsorship information is necessary to fulfill M.I.T. obligations under research contracts.

5. If no contract or grant, was there significant use of M.I.T. administered funds or facilities as defined in Instructions? YES ☐ NO ☑

6. DATES OF CONCEPTION and PUBLIC DISCLOSURE (accurate data is essential as prior disclosure may affect the possibility of obtaining patent rights)	DATE	REFERENCES/COMMENTS Please include names of periodicals/journals. (use separate sheet if necessary)
A. Date of conception of invention. Has this date been documented? If so, where?		DOCUMENTED IN LABORATORY NOTEBOOK
B. First publication containing sufficient description to enable a person skilled in this field to understand and to make or use the invention. (include theses, and the date submitted)	-	RECEIVED
C. First public oral disclosure of invention sufficient to enable a person skilled in this field to understand and to make or use the invention.	-	-
D. If unpublished and undisclosed, provide the anticipated publication or oral disclosure date and any submissions made for potential publication.		

7. Has the invention been reduced to practice? YES ☑ NO ☐ If yes, please give date of first reduction to practice

8. Please attach list of any commercial entities that may be interested in this invention. (provide as much detail as possible)

9. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true.

I (We) hereby agree to assign all right, title and interest to this invention to M.I.T. and agree to execute all documents as requested, assigning to M.I.T. our rights in any patent application filed on this invention, and to cooperate with the M.I.T. Technology Licensing Office in the protection of this invention. M.I.T. will share any royalty income derived from the invention with the inventor(s) according to its standard policies.

Inventor's Signature: Klavs Jensen Date
Home Address: 103 OUTLOOK DRIVE, LEXINGTON, MA 02421
Social Security No. (required): 396-78-3412 Country of Citizenship: US

Inventor's Signature: Saif Khan Date
Home Address: 505 MEMORIAL DRIVE, #603A, CAMBRIDGE, MA 02139
Social Security No. (required): 030-84-7784 Country of Citizenship: INDIA

saifkhan@mit.edu

Inventor's Signature Date
Home Address
Social Security No. (required) Country of Citizenship

Inventor's Signature Date
Home Address
Social Security No. (required) Country of Citizenship

Please note that Social Security number and country of citizenship are required; absence of this information may hinder distribution of the inventors' share of any royalties that may result from this technology. If there are more than four inventors, please attach additional form

Technology disclosed to and understood by:
Signature of Non-Inventor Witness: Axel Günther Date
Name and Title of Witness (please type or print): AXEL GUENTHER, DR.

(1/15/2002)

Microchemical Systems for Synthesis and Coating of Colloidal Nanoparticles

General purpose

We have conceptualized, designed and demonstrated novel microfluidic chemical systems for synthesis and coating of colloidal nanoparticles. These systems will be able to accomplish continuous synthesis of monodisperse particles and *in situ* coating of their surfaces with various functionalities, through novel reactant-contacting schemes.

Colloidal nanoparticles have innumerable applications in almost all fields of science, and are ubiquitous in materials science, chemistry and biology. Industrial applications of colloidal spheres of silica and titania, for example, include adhesion and lubrication technology, pigments, catalysis, thin films for photovoltaic, electrochromic, photochromic, electroluminescent devices, sensors, foods, health-care, anti-reflective coatings, chromatography, ceramics, optoelectronics, photonic band-gap (PBG) materials etc. Even more fascinating are the applications of these particles when their surfaces are modified or coated in some manner by other functionalities. Such 'nanocomposites' find numerous applications in fields ranging from opto-electronics and lasers to drug-delivery and biotechnology.

Technical description

Synthesis of colloidal particles of silica (SiO_2) is accomplished in a microreactor depicted in Figure 1. The microreactor fabricated in poly-dimethyl siloxane (PDMS) consists of a micromixing section followed by an ageing section where the particles grow to their final sizes. Sol-gel chemistry is employed, with the silicon precursor being Tetraethyl orthosilicate (TEOS). The results of Titania (TiO_2) particle synthesis, and a comparison with particles obtained via conventional methods clearly indicate the advantages of processing in microreactors for enhanced particle quality.

We have also designed novel microfluidic devices whereby the colloidal particles synthesized in the manner described above are coated with other substances. The fabrication of these devices is currently under progress. The basic requirement for on-chip, *in situ* coating is the ability to transfer the colloidal particles from one stream to another. Our devices accomplish this objective by means of the novel concept of 'electrophoretic switches'. Figure 3 illustrates this concept in more detail. The colloidal particles, as synthesized, are charged. The solvent stream containing the particles is contacted with another stream of pure solvent, between a pair of gold electrodes. By manipulating the potential applied to these electrodes, it is possible to transport the colloidal particles from the first stream to the pure solvent stream by the phenomenon of electrophoresis. The two streams are then separated at the exit of the device. The small dimensions of the microfluidic device ensure laminar flow, and thus very little mixing between streams. Fabrication of these devices is achieved through techniques developed in our laboratory.

A composite device consisting of two electrophoretic switches and an intermediate ageing length section is illustrated in Figure 4. In this device, the particles are switched from the solvent stream to a stream containing the reactants for surface coating. The ageing section provides equilibration length, and the switch at the exit transfers the coated particles into a pure solvent stream. By controlling flow rates, and hence residence times in the ageing section, it is possible to obtain coatings of desired thicknesses.

Advantages over existing methods

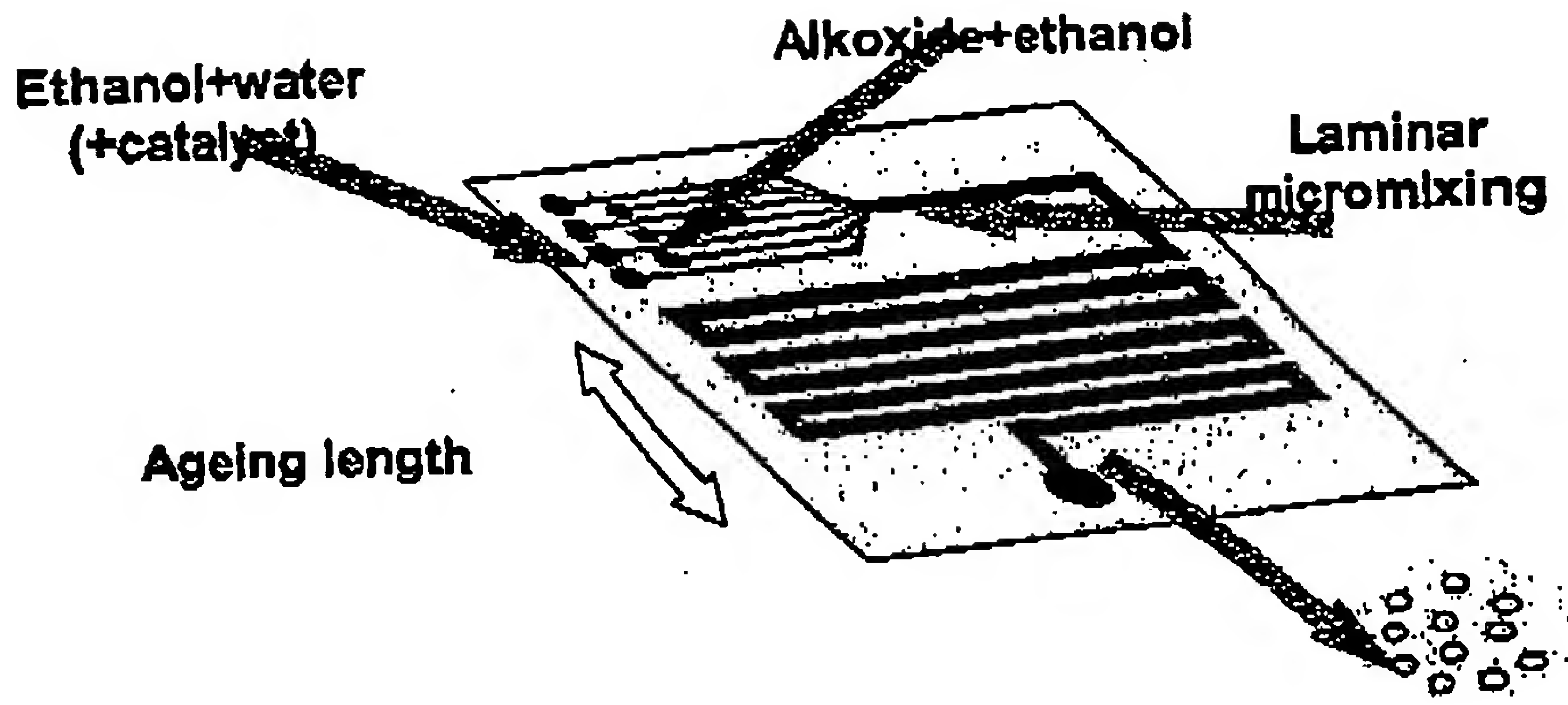
Our devices represent a radical departure from most conventional macro-scale batch processing methods employed to synthesize and coat colloidal nanoparticles. The main difference is that synthesis and coating are *in series* and *in situ*. This obviates the need for numerous cumbersome, and often expensive, intermediate-processing steps. These steps involve multiple washings and centrifugations, and often degrade particle quality. In addition, the tremendous degree of control over the physical transport phenomena afforded by microfluidic devices will enable us to accurately tailor particle morphology and surface nature in our devices. As shown in the previous section, the quality of particles obtained from our microreactor is much better than those obtained from conventional methods of synthesis, primarily due to the controlled fluidic environment inside the microchannels. By operating multiple devices in parallel, it is possible to rapidly obtain information on a wide array of synthesis and coating chemistries, thereby also enabling a combinatorial materials screening platform.

Our approach

We can

Commercial Applications

As noted in the introduction, there is a huge list of applications where coated colloidal nanoparticles could be used. The availability of a platform that could rapidly, easily synthesize and coat particles exactly to specifications would be of immense interest to a variety of industries. In the chemical industry, these coated colloids would be of interest due to their enhanced catalytic properties. In the materials industry, these would be of interest as building blocks for a wide array of interesting optical materials, including photonic-band gap materials and optical filters. Applications in the biotechnology and drug-delivery industry would include controlled coating and immobilization of biological macromolecules (proteins, enzymes etc) on the surfaces of colloidal particles, which could then act as controlled drug delivery vectors.



Dimensions

- Inlet channels: 50 µm
- Ageing channels: 400 µm
- Channel depth: 50 µm
- Total ageing length: 90 cm
- Flow rates: 5-20 µl/min
- Linear velocities: 4.2-16.8 mm/sec

Figure 1: Layout of microreactor for synthesis of colloidal nanoparticles via sol-gel processing.

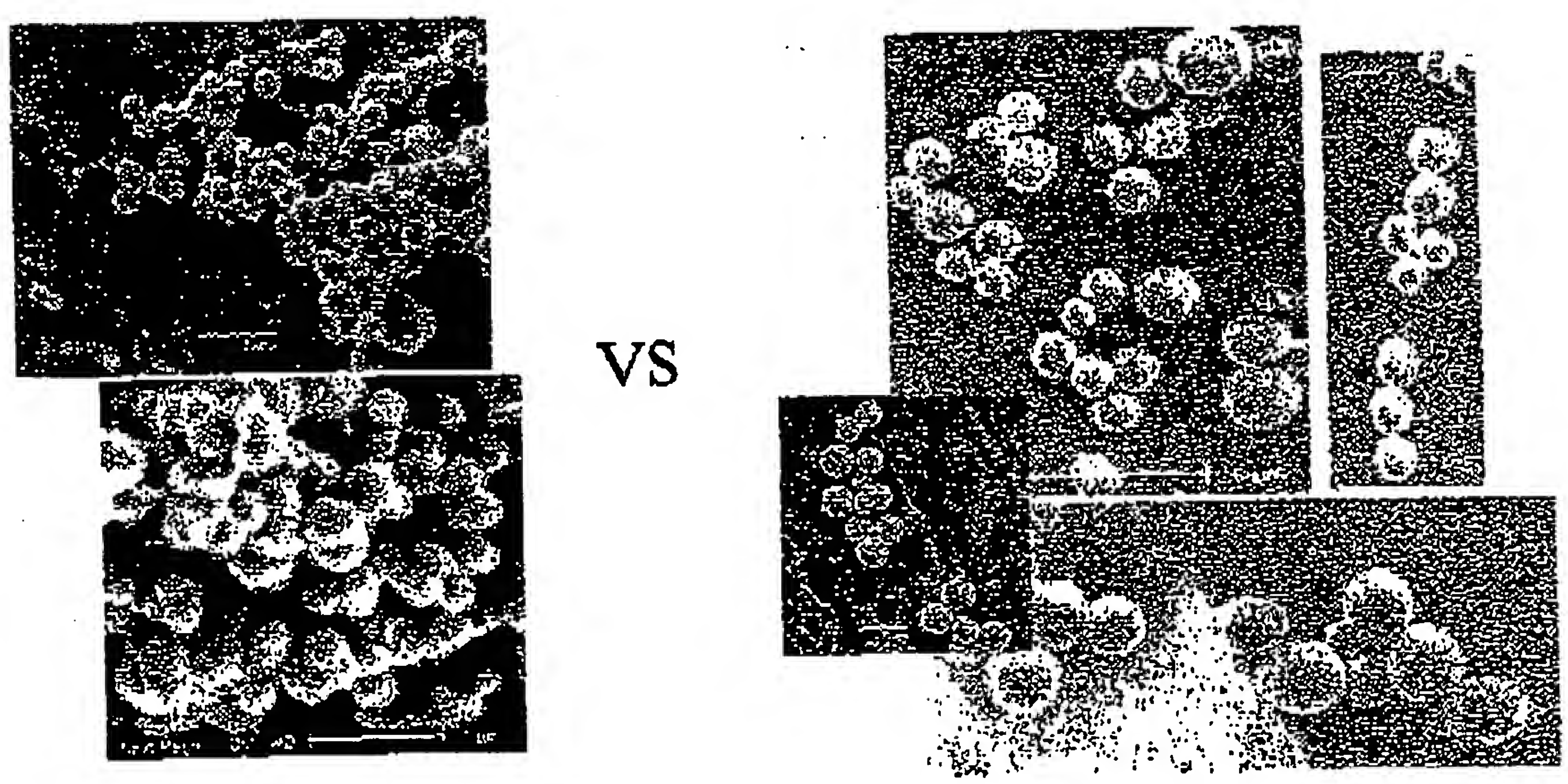


Figure 2: Comparison of TiO_2 particles obtained via conventional methods (left) and microreactor (right).

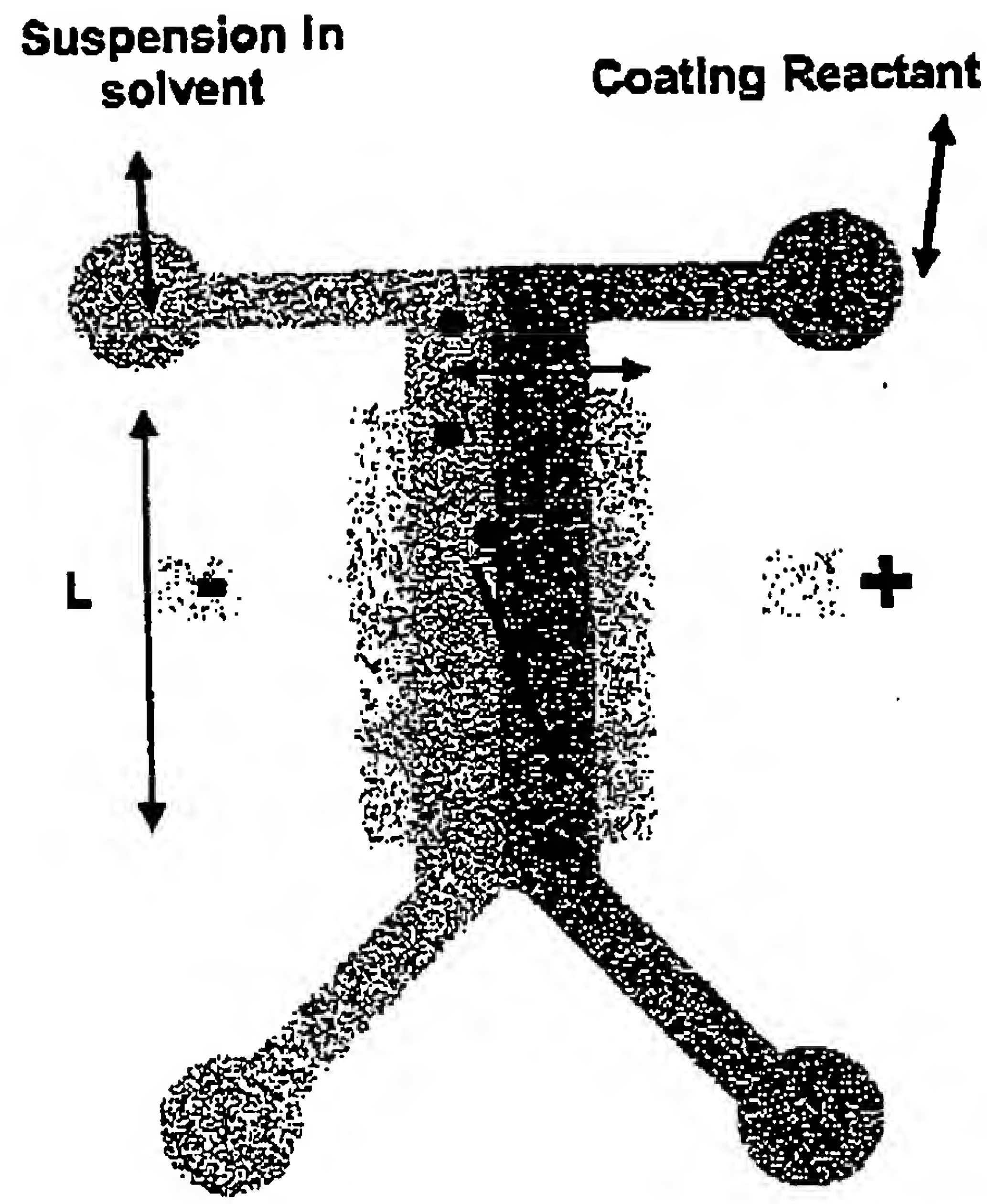


Figure 3: Concept of Electrophoretic Switch

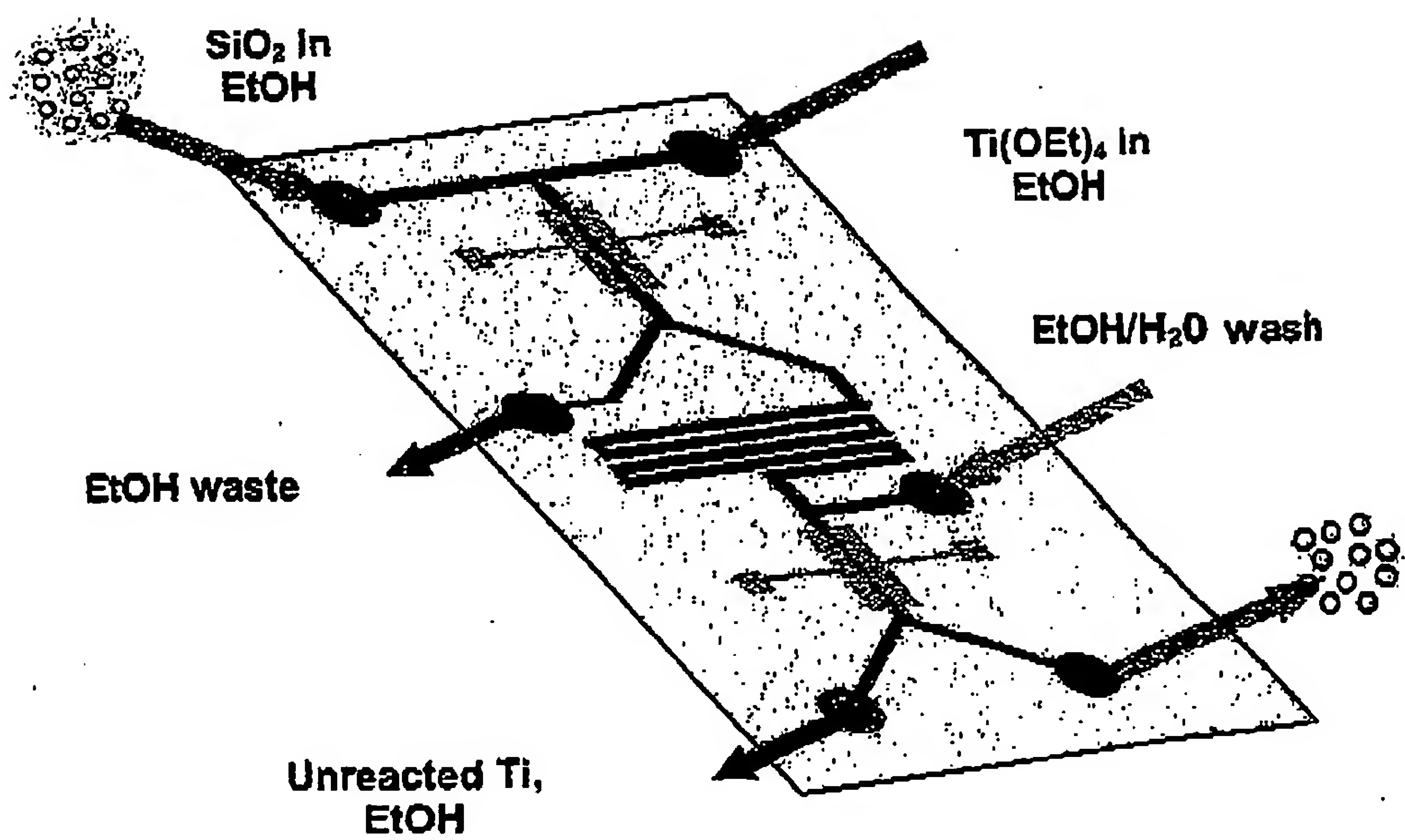


Figure 4: Illustration of composite device

Exhibit B

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Mr. Bo Pasternack
Choate, Hall & Stewart
Exchange Place, 34th floor
53 State Street
Boston, MA 02109-2804

RE: M.I.T. Case No. 10051 "Microchemical Systems for Synthesis and Coating of Colloidal Nanoparticles"

Dear Bo:

Enclosed is the invention disclosure for the above-referenced case. Please provide an estimate for filing a utility application. Also enclosed is a Task Initiation Form which should be completed and returned to my attention.

If you have any questions or need any additional information, please contact Steve. Thanks so much.

Regards,

Maryann B. Kabarsky
Assistant to Steve Brown

Enc.